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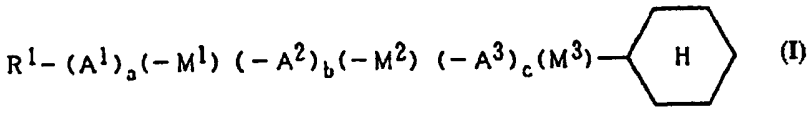
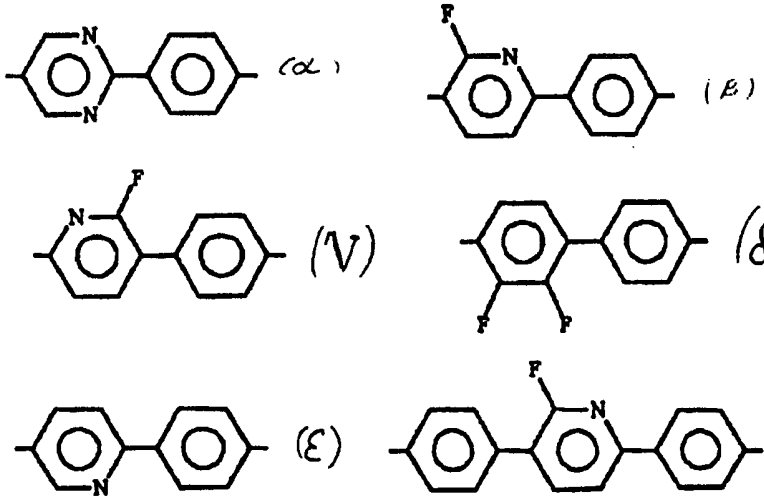


(I)



(57) Abstract

The present invention provides compounds of formula (I) and liquid-crystal compositions containing the compounds, in which R^1 is, for example, a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, in which one $-CH_2-$ group may be replaced by -O- or -CO-O-; and the group $-(A^1)_a(-M^1)(-A^2)_b(-M^2)(-A^3)_c(M^3)-$ is any one of following groups : (α), (β), (γ), (δ), (ε), (φ). The compound of the present invention is useful as a component of smectic or ferroelectric liquid-crystal compositions having a broad temperature range of a smectic C phase, particularly having a broad temperature range in a lower temperature region.

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Novel Compounds for Use in Liquid-Crystal Compositions

The present invention relates to a novel compound for use in liquid-crystal compositions.

In particular in the last decade, liquid crystals have been introduced into various technical areas where electro-optical and display device properties are required (for example, in watch, calculator and typewriter displays). These display devices are based on the dielectric alignment effects in the nematic, cholesteric and/or smectic phases of the liquid-crystalline compounds, where, caused by the dielectric anisotropy, the molecular long axes of the compounds adopt a preferred alignment in an applied electric field. The conventional response times in these display devices are too long for many other potential areas of application of liquid crystals. This disadvantage is particularly noticeable if a large number of pixels have to be addressed. The production costs of equipment containing relatively large screen areas are then generally too high.

In addition to nematic and cholesteric liquid crystals, optically active smectic liquid-crystal phases have also been increasing in importance for a few years.

Clark and Lagerwall have been able to show that the use of ferroelectric liquid-crystal systems in very thin cells give electro-optical switch or display elements which have response times faster by a factor of up to 1000 compared with conventional TN ("twisted nematic") cells (cf., for example, Lagerwall et al., "Ferroelectric Liquid Crystals for Displays", SID Symposium, October Meeting 1985, San Diego, Ca., USA). Due to these and other favorable properties, for example, the possibility for bistable switching and the contrast which is virtually independent of the viewing angle, FLCs are fundamentally very suitable for the above-mentioned

areas of application, for example, via matrix addressing. Due to their high contrast and speed, ferroelectric liquid crystals are also particularly suitable in the area of spatial light modulators (cf, for example, U. Efron in "Spatial Light Modulators and Applications", SPIE, Vol. 1150, p. 46 ff).

Ferroelectric properties are expressed in liquid crystals having a phase called a smectic phase represented by a smectic C phase. Compounds having a smectic phase are described in, for example, G. W. Gray et al., Mol. Cryst. Liq. Cryst., Vol. 37, 157-188 (1976). The working temperature and the storage temperature of such liquid-crystal display devices are restricted by the temperatures range of a smectic C phase. Hence ferroelectric liquid crystals having a smectic C phase in a temperature range of -20 °C to +70 °C are desired.

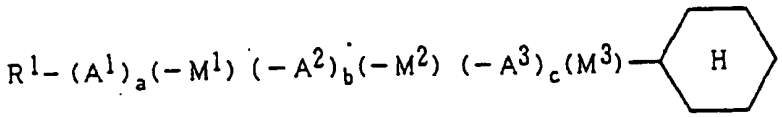
The above requirements can only be satisfied by mixing a plurality of components. In addition, a material which can reduce the melting point and raise the upper limit of the phase transition temperature of a smectic C phase is always required. Compounds bearing a very short alkyl or alkyloxy radical on only one side of the molecule tend to have a nematic phase. It has been an established technique in the art to use such compounds as a component of nematic liquid crystal compositions (Japanese Patent Public Disclosure No. 148282/1986 and No. 003451/1980). However, they have not been considered to be useful for ferroelectric liquid-crystal compositions.

Accordingly, the object of the present invention is to provide a compound capable of broadening the temperature range of a smectic C phase and to provide a liquid-crystal composition containing the compounds.

Surprisingly, it has now been found that compounds which include as a terminal group a cyclohexyl group having no side chains have a smectic C phase and are useful for broadening

the temperature range of the smectic C phase and reducing the melting point of smectic liquid-crystal compositions and ferroelectric liquid crystals.

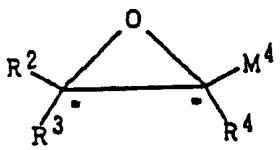
The present invention relates to compounds of the formula (I):

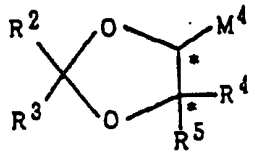


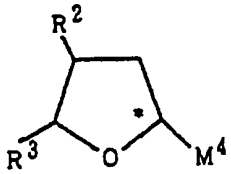
(I)

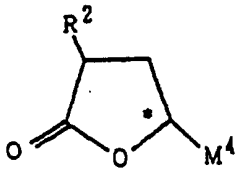
in which the symbols and indices have the following meanings:

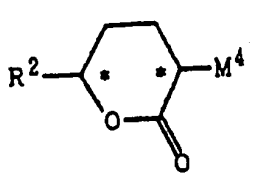
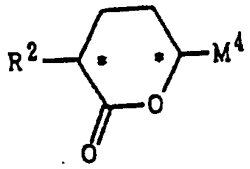
R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, and in which, in addition, one or more hydrogen atoms of the alkyl radical may be substituted by F, Cl, Br or CN, or is one of the chiral groups below:

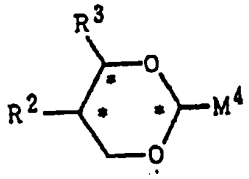
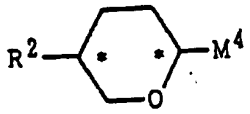


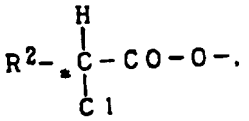
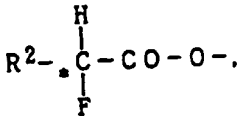


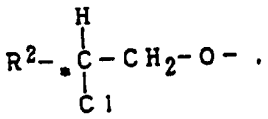
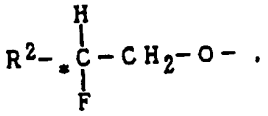


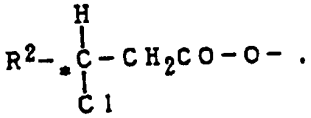
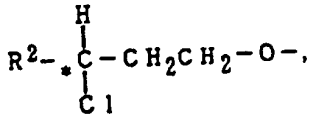


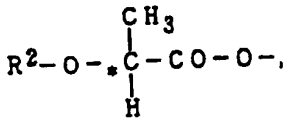
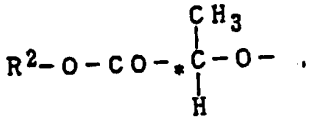



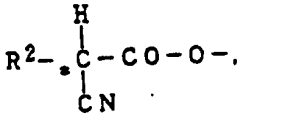
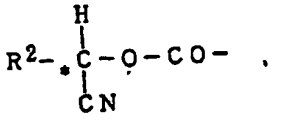



wherein,

R^2, R^3, R^4 and R^5, independently of one another, are H or a straight-chain or branched alkyl radical having 1 to 22 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, or R^2 and R^3 together may alternatively be $-(CH_2)_4-$ or $-(CH_2)_5-$ if they are bonded as substituents to a dioxolane system; and

M^4 is $-CH_2-O-$, $-O-CH_2-$, -CO-O-, -O-CO- or a single bond;

A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, in which one or two hydrogen atoms may be replaced by F, Cl and/or CN, pyrazine-2,5-diyl, pyridazine-3,6-diyl, pyridine-2,5-diyl or pyrimidine-2,5-diyl, in which one or two hydrogen atoms may be replaced by F, trans-1,4-cyclohexylene, in which one or two hydrogen atoms may be replaced by -CN and/or $-CH_3$, 1,3,4-thiadiazole-2,5-diyl, 1,3-dioxane-2,5-diyl, 1,3-dithiane-2,5-diyl, 1,3-thiazole-2,4-diyl, 1,3-thiazole-2,5-diyl, thiophene-2,4-diyl, thiophene-2,5-diyl, piperazine-1,4-diyl, piperazine-2,5-diyl or naphthalene-2,6-diyl;

M^1 and M^2 are identical or different and are a single bond, -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

M^3 is a single bond or a straight-chain or branched alkyl radical having 1 to 16 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C- or $-Si(CH_3)_2-$, and in which, in addition, one or more hydrogen atoms of the alkyl radical may be substituted by F, Cl, Br or CN, with the proviso that M^3 is not $-O-CO-CH_2CH_2-$;

a, b and c are zero or one, with the proviso that the

sum a+b+c is 2 or 3; and

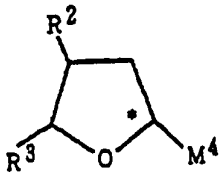
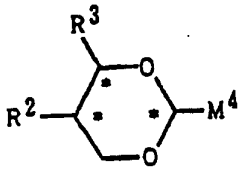
* is a chiral center;

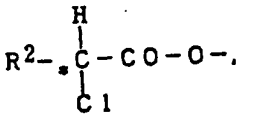
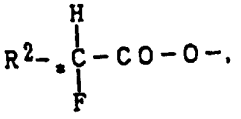
provided however that when A^1 and A^2 are 1,4-phenylene, M^1 and M^2 are a single bond, c is zero and M^3 is -CO-O-, then R^1 is not C_8H_{17}-O-.

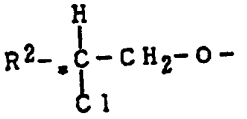
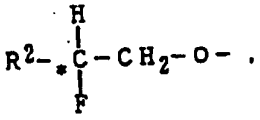
The present invention further relates to liquid-crystal compositions containing at least one compound of the formula (I) or a mixture thereof.

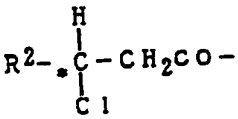
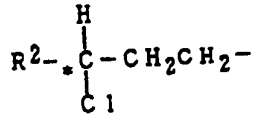
Preference is given to the compounds of the formula (I) in which the symbols and indices have the following meanings:

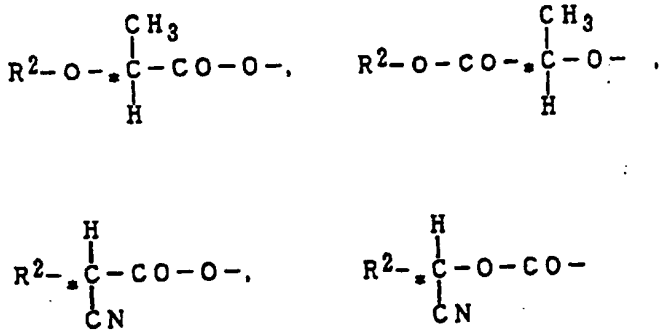
R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -O-CO-O-, -CH=CH-, -C≡C-, , or $-Si(CH_3)_2-$, or is one of the chiral groups below:



wherein,

R^2, R^3, R^4 and R^5, independently of one another, are H or a straight-chain or branched alkyl radical having 1 to 22 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, or R^2 and R^3 together may alternatively be $-(CH_2)_4-$ or $-(CH_2)_5-$ if they are bonded as substituents to a dioxolane system;

A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, in which one or two hydrogen atoms may be replaced by F, pyrazine-2,5-diyl, pyridazine-3,6-diyl, pyridine-2,5-diyl, pyrimidine-2,5-diyl, trans-1,4-cyclohexylene, 1,3,4-thiadiazole-2,5-diyl, 1,3-dioxane-2,5-diyl or naphthalene-2,6-diyl;

M^1 and M^2 are identical or different and are a single bond, -O-, -CO-, -CO-O-, -O-CO-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

M^3 is a single bond or a straight-chain or branched alkyl radical having 1 to 16 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -CO-, -CO-O-, -O-CO- or -O-CO-O-, and in which, in addition, one or more hydrogen atoms of the alkyl radical may be substituted by F, Cl, Br or CN; and

a, b and c are zero or one, with the proviso that the sum a+b+c is 2 or 3.

Particular preference is given to the compounds of the formula (I) in which symbols and indices have the following meanings:

R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -CO-, -CO-O-, -O-CO-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$;

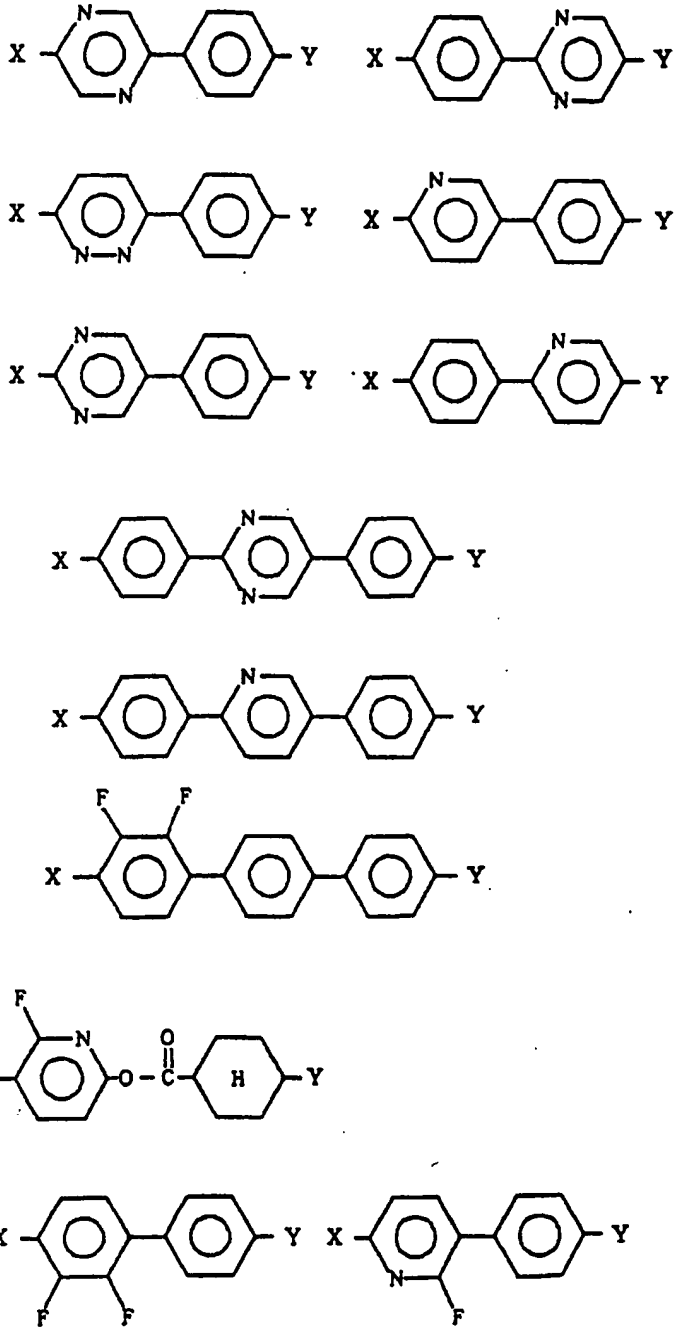
A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, in which one or two hydrogen atoms may be replaced by F, pyrazine-2,5-diyl, pyridine-2,5-diyl, pyrimidine-2,5-diyl, trans-1,4-cyclohexylene, 1,3,4-thiadiazole-2,5-diyl, naphthalene-2,6-diyl or 1,3-dioxane-2,5-diyl;

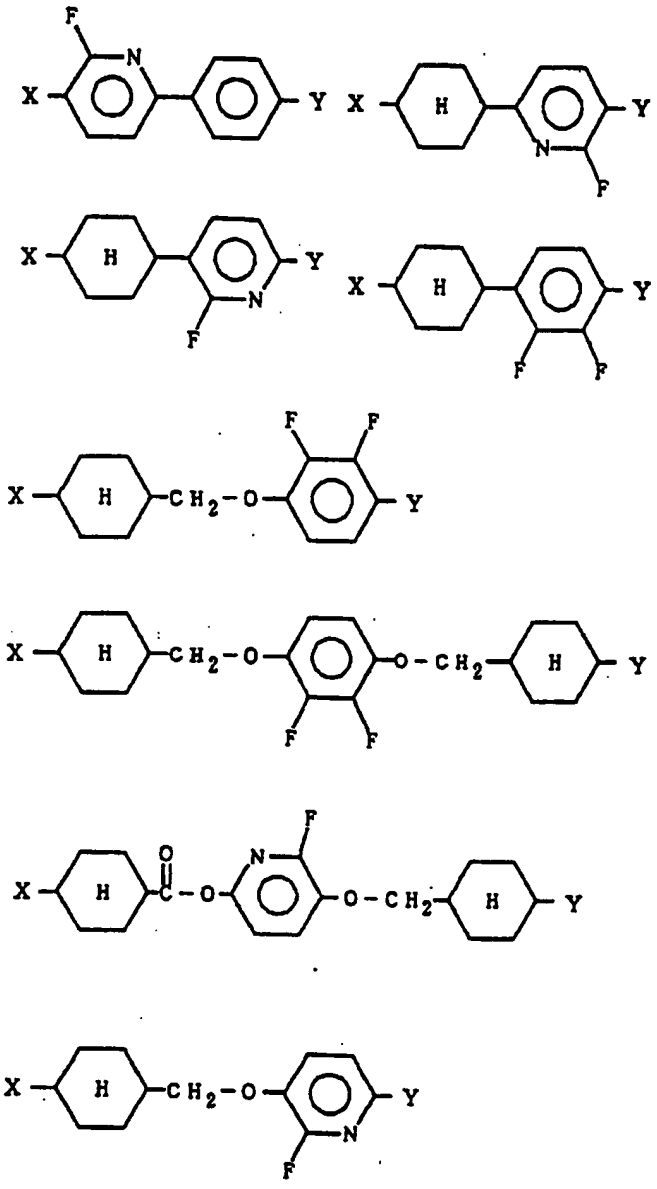
M^1 and M^2 are identical or different and are a single bond, -O-, -CO-O-, -O-CO-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

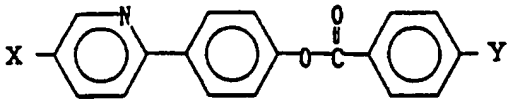
M^3 is a single bond, -CO-O-, -O-CO-, $-O-CO-C_nH_{2n}-$ or $-O-C_nH_{2n}-$ (wherein n is an integer from 1 to 10); and

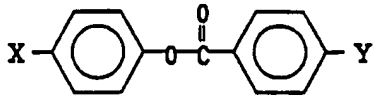
a, b and c are zero or one, with the proviso that the sum a+b+c is 2 or 3.

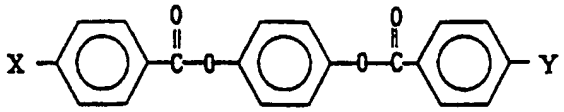
Of the compounds of the formula (I) according to the present invention, the followings are particularly preferable:

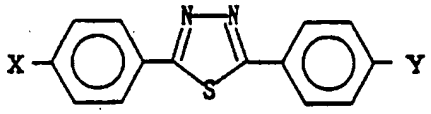


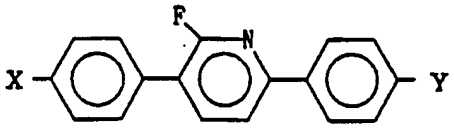


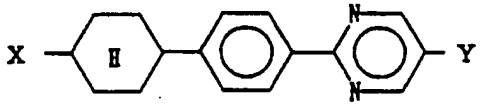

X
N
O
C
O
Y

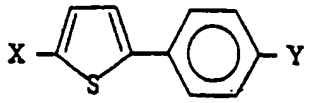

X
O
C
O
Y

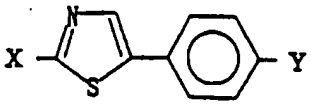

X
O
C
O
C
O
Y

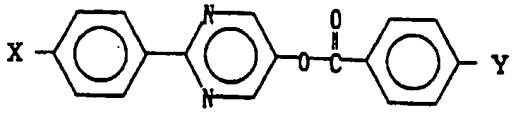

X
N
N
S
Y

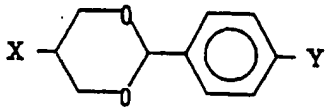

F
N
X
Y

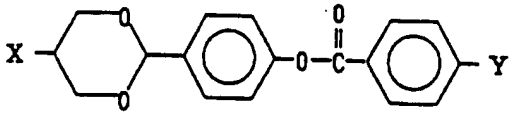

X
H
N
N
Y

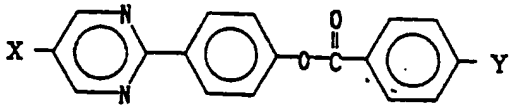






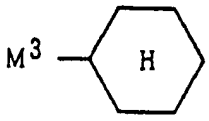






in which either of X and Y is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, wherein one $-CH_2-$ group may be replaced by -O-,  , -CH=CH-, -CO-O- or $-Si(CH_3)_2-$; and

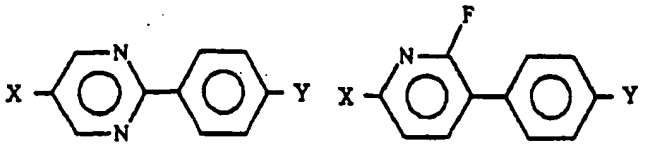
the other is

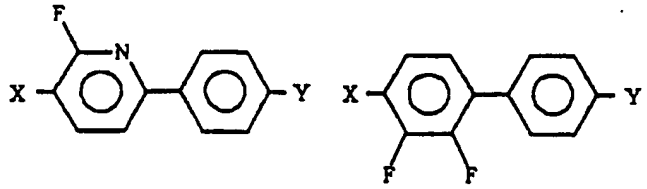


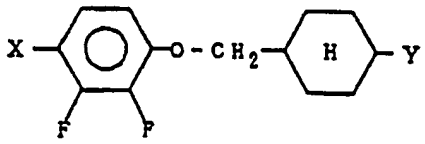
wherein M^3 is a single bond, -CO-O-, -O-CO-, $-O-CO-C_nH_{2n}-$ or $-O-C_nH_{2n}-$ (wherein n is an integer from 1 to 10).

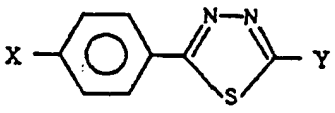
Very particular preference is given to the following

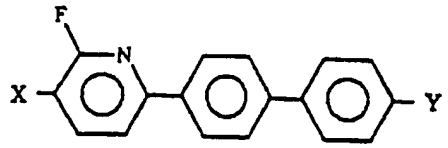
compounds:

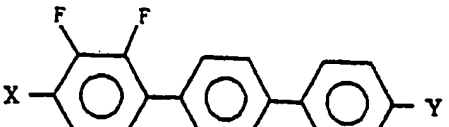


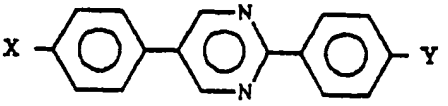






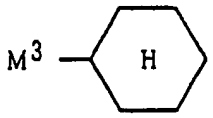






in which either of X and Y is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, wherein one $-CH_2-$ group may be replaced by -O- or -CO-O-; and

the other is

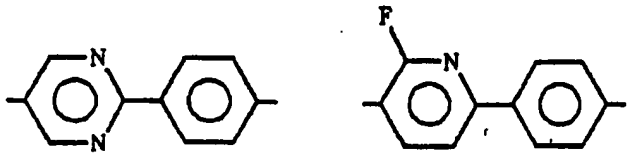


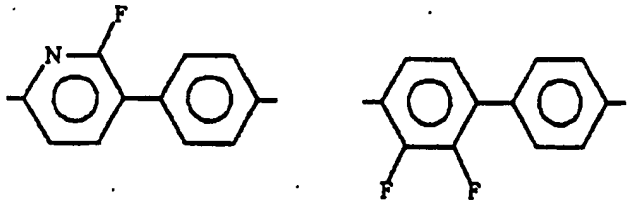
wherein M^3 is -O-CO- or $-O-CH_2-$,

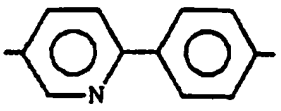
and the compounds of the formula (I) wherein:

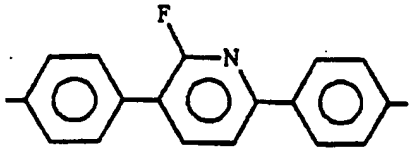
R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, in which one $-CH_2-$ group may be replaced by -O- or -CO-O-; and

the group $-(A^1)_a(-M^1)(-A^2)_b(-M^2)(-A^3)_c(-M^3)-$ is any one of the following groups:









The compound of the formula (I) according to the present invention is suitable as a component for liquid-crystal compositions, in particular ferroelectric liquid-crystal compositions. The liquid-crystal compositions may contain from 0.01 to 60 % by weight, preferably from 0.1 to 40 % by weight, particularly preferably from 0.1 to 20 % by weight, of the compounds according to the present invention. The other constituents are preferably selected from known compounds having nematic, cholesteric and/or smectic phases; these include, for example, Schiff's bases, biphenyls, terphenyls, phenylcyclohexanes, cyclohexylbiphenyls, N-, S- or O-containing heterocyclic compounds, for example pyrimidines, cinnamic acid esters, cholesterol esters or various bridged, polycyclic esters of p-alkylbenzoic acids which have terminal polar groups.

Surprisingly, it has now been found that the addition of compounds of the formula (I) can considerably broaden the temperature range in a lower temperature region of smectic C compositions or the smectic C phase of ferroelectric liquid-crystal compositions.

These mixtures can in turn be used in electro-optical or fully optical elements, for example, display elements, switching elements, light modulators, elements for image processing, signal processing or generally in the area of non-linear optics.

The present invention is described in greater detail by means of Examples below.

The phase transition temperatures were determined with the aid of a polarizing microscope from the changes in texture on heating. By contrast, the melting point was determined using a DSC instrument. The phase transition temperatures between the phases

isotropic	(I)
nematic	(N or N*)
smectic-C	(S_c or S_c*)

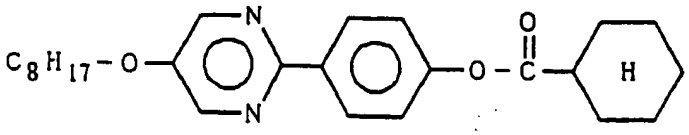
smectic-A (S_A)

crystalline (X)

are given in °C, and the values are between the phase designations in the phase sequence.

Example 1

4-(5-Octyloxypyrimidine-2-yl)phenyl cyclohexylcarboxylate



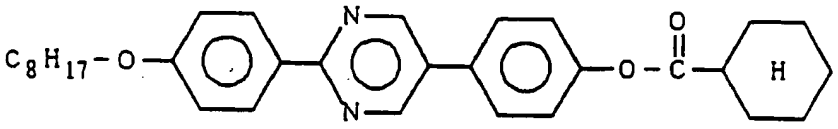
1.00 g of 4-(5-octyloxypyrimidine-2-yl) was dissolved in 12 ml of pyridine, and the solution was cooled on an ice bath. 4 g of cyclohexyl carbonylchloride was slowly added dropwise to the solution. The solution was left at room temperature overnight and poured into ice water. After the solution was adjusted to pH 2 with concentrated hydrochloric acid, an ester was precipitated. The solution was filtered and recrystallized from n-hexane to give 600 mg of 4-(5-octyloxypyrimidine-2-yl)phenyl cyclohexylcarboxylate.

The compound has the following phase sequence:

X 107 I

Example 2

4-[2-(4'-Octyloxyphenyl)pyrimidine-5-yl]phenyl cyclohexylcarboxylate



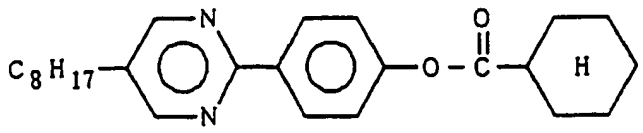
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 154.9 I

Example 3

4-(5-Octylpyrimidine-2-yl)phenyl cyclohexylcarboxylate



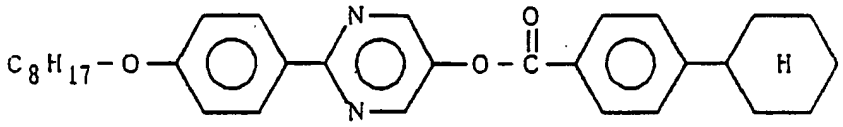
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 96.7 I

Example 4

2-(4-Octyloxyphenyl)pyrimidine-5-yl
4-cyclohexylphenylcarboxylate



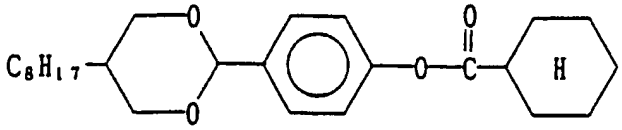
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 108 (73) N 206 I

Example 5

4-(5-Octyl-1,3-dioxane-2-yl)phenyl cyclohexanecarboxylate



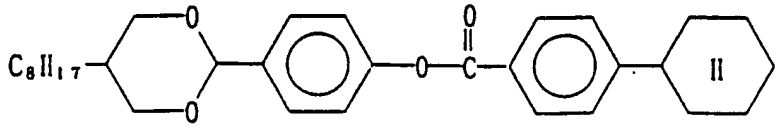
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 82 (22.5) S_2 61 S_1 75 I

Example 6

4-(5-Octyl-1,3-dioxane-2-yl)phenyl
4-cyclohexylphenylcarboxylate

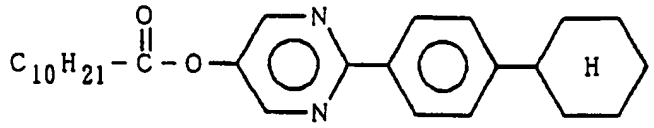


The synthesis was carried out analogously to Example 1.
The compound has the following phase sequence:

X_2 99 X 106 (78) N 169 I

Example 7

2-(4-Cyclohexylphenyl)pyrimidine-5-yl undecanoate

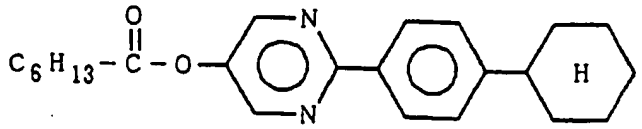


The synthesis was carried out analogously to Example 1.
The compound has the following phase sequence:

X 99.9 (88) I

Example 8

2-(4-Cyclohexylphenyl)pyrimidine-5-yl heptanoate

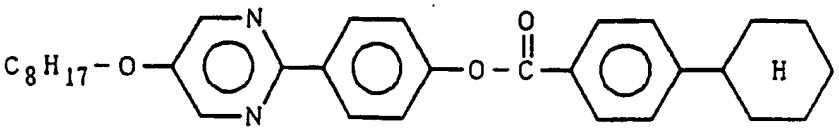


The synthesis was carried out analogously to Example 1.
The compound has the following phase sequence:

X 109.3 (100) I

Example 9

4-(5-Octyloxypyrimidine-2-yl)phenyl 4-cyclohexylphenylcarboxylate



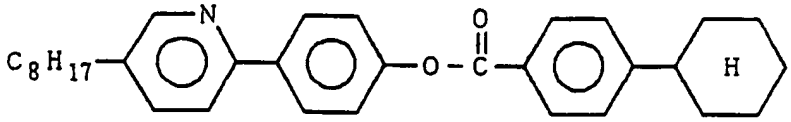
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 144 (122) N 200 I

Example 10

4-(5-Octylpyridine-2-yl)phenyl 4-cyclohexylphenylcarboxylate



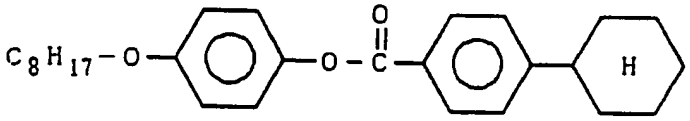
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 133 (117) N 182 I

Example 11

4'-Octyloxylphenyl 4-cyclohexylbenzoate



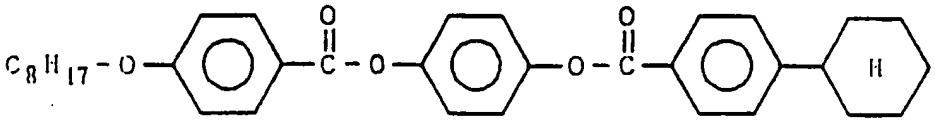
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 92 (78) I

Example 12

Hydroquinone 4-octyloxyphenylcarboxylate 4-cyclohexylphenylcarboxylate diester



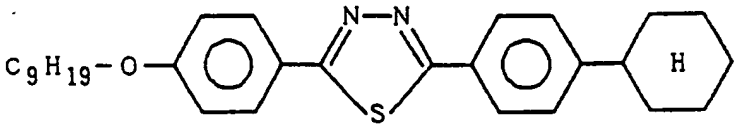
The synthesis was carried out analogously to Example 1.

The compound has the following phase sequence:

X 146 (114) N 212 I

Example 13

2-(4-Cyclohexylphenyl)-5-(4-nonyloxyphenyl)-1,3,4-thiadiazole



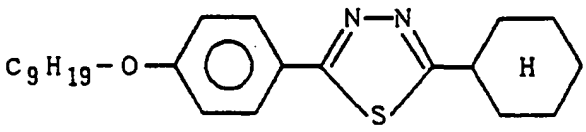
The synthesis was carried out analogously to the method described by K. Dimitrooa, F. Hauschild, H. Azschke and H. Schubert in Journal für Prakt. Chemie, Vol. 322 (1980), page 933.

The compound has the following phase sequence:

X 126 S_c 146 N 186 I

Example 14

2-Cyclohexyl-5-(4-nonyloxyphenyl)-1,3,4-thiadiazole



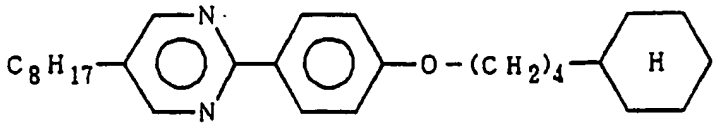
The synthesis was carried out analogously to Example 13.

The compound has the following phase sequence:

X_2 77 X 86 S_A 67 I

Example 15

2-[4'-(4-Cyclohexyl-n-butyloxy)phenyl]-5-octylpyrimidine



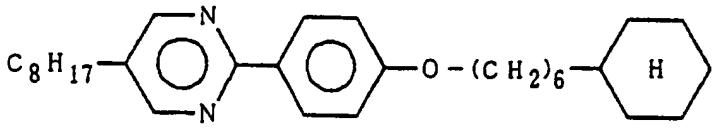
The synthesis was carried out according to the method described in European Patent No. 0318423 and No. 0398155.

The compound has the following phase sequence:

X 42 (11) S_c 41 S_A 43 N 51 I

Example 16

2-[4-(6-Cyclohexyl-n-hexyloxy)phenyl]-5-octylpyrimidine



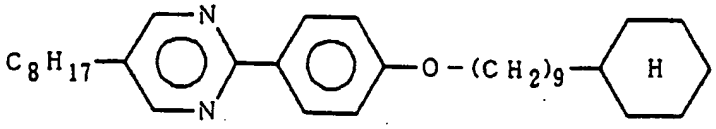
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 56 (10) S_c 51 S_A 52 N 57 I

Example 17

2-[4'-(9-Cyclohexyl-n-nonyloxy)phenyl]-5-octylpyrimidine



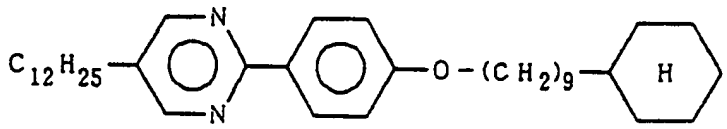
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 73 (67) N 60 I

Example 18

2-[4'-(9-Cyclohexyl-n-nonyloxy)phenyl]-5-dodecylpyrimidine



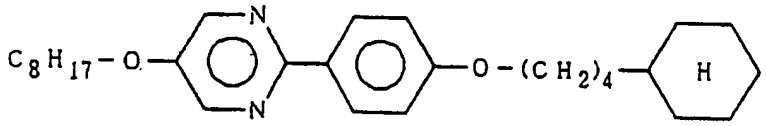
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 78 (65) S_c 75 I

Example 19

2-[4-(4-Cyclohexylbutyloxy)phenyl]-5-octyloxypyrimidine



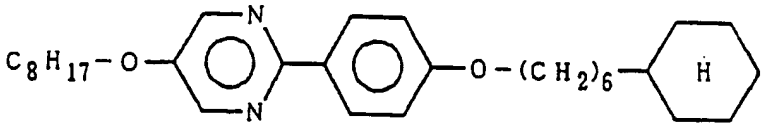
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 71 (51) S_c 79 S_A 81 N 84 I

Example 20

2-[4-(4-Cyclohexylhexyloxy)phenyl]-5-octyloxypyrimidine



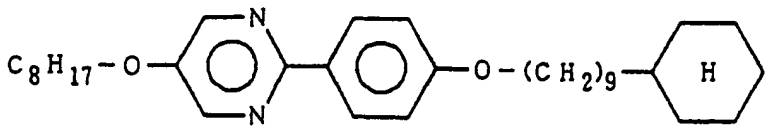
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 55 (41) S_c 81 S_A 87 N 88 I

Example 21

2-[4-(9-Cyclohexyl-n-nonyloxy)phenyl]-5-octyloxypyrimidine



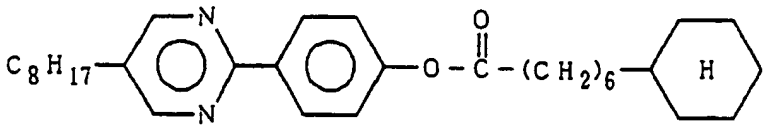
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 73 (61) S_c 86 S_A 91 I

Example 22

4-(5-Octylpyrimidine-2-yl)phenyl 7-cyclohexylheptoate



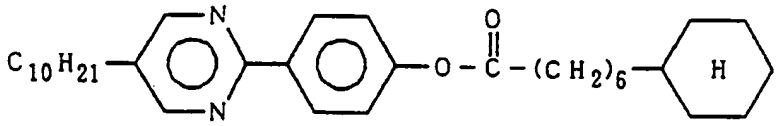
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X_2 69 X 71 I

Example 23

4-(5-Decylpyrimidine-2-yl)phenyl 7-cyclohexylheptoate



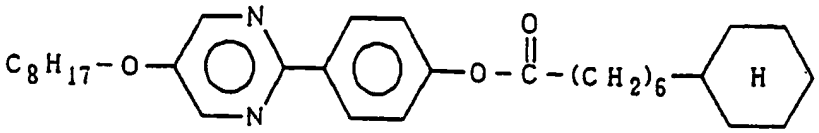
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X_2 62 (56) X 74 S_c 64 I

Example 24

4-(5-Octyloxypyrimidine-2-yl)phenyl 7-cyclohexylheptoate



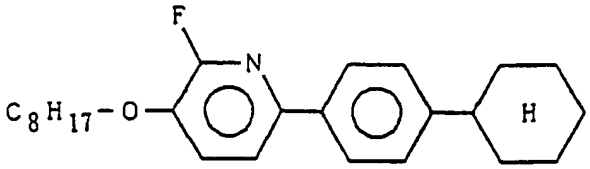
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 66 (58) S_c 87 S_A 88 N 90 I

Example 25

6-(4-Cyclohexylphenyl-2-fluoro-3-octyloxypyridine)



2.2 g (8.3 mmol) of triphenylphosphine and 1.3 ml (8.3 mmol) of diethyl azodicarboxylate were stirred in tetrahydrofuran at 0 °C for half an hour. 1.3 ml (8.3 mmol) of 1-octanol and 1.5 g (5.53 mmol) of 6-(4-cyclohexylphenyl)-2-fluoro-3-hydroxypyridine were added therein and the mixture was stirred at room temperature for 18 hours.

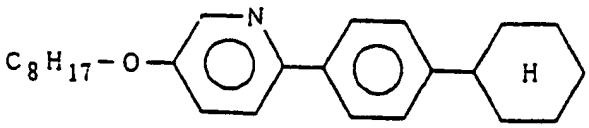
The solution was evaporated to dryness, and the residue was purified by column chromatography. Recrystallization from acetonitrile gave 1.03 g of the titled compound.

The compound has the following phase sequence:

X 86 (65) I

Example 26

2-(4-Cyclohexylphenyl)-octyloxypyridine

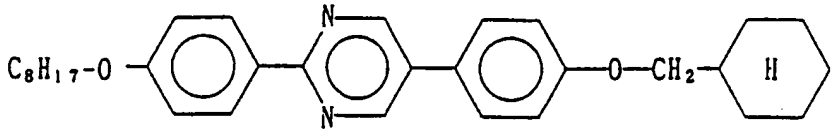


The synthesis was carried out analogously to Example 25.
The compound has the following phase sequence:

X 68 (47) S_2 75 S_c 84 N 89 I

Example 27

5-[4-(Cyclohexylmethyloxy)phenyl]-2-(4'-octyloxyphenyl)-pyrimidine

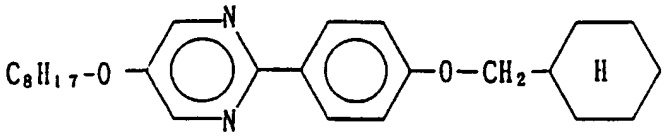


The synthesis was carried out analogously to Example 25.
The compound has the following phase sequence:

X 126 S_3 140 S_c 183 S_A 213 N 214 I

Example 28

2-[4-(Cyclohexylmethyloxy)phenyl)-5-octyloxypyrimidine

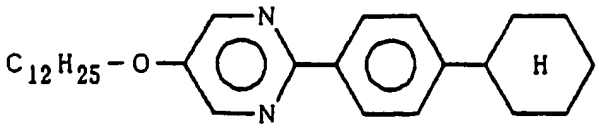


The synthesis was carried out analogously to Example 25.
The compound has the following phase sequence:

X 106 I

Example 29

2-(4-Cyclohexylphenyl)-5-dodecyloxypyrimidine

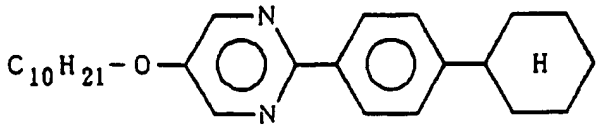


The synthesis was carried out analogously to Example 25.
The compound has the following phase sequence:

X_1 67 (32) X_2 71 (39) S_c 75 S_A 92 I

Example 30

2-(4-Cyclohexylphenyl)-5-decyloxypyrimidine

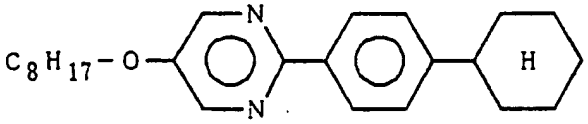


The synthesis was carried out analogously to Example 25. The compound has the following phase sequence:

X 65 (23) S_3 74 S_C 76 S_A 89 N 89.2 I

Example 31

2-(4-Cyclohexylphenyl)-5-octyloxypyrimidine

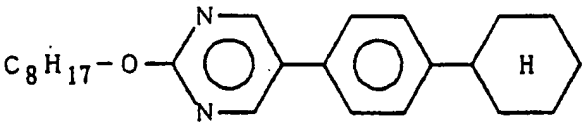


The synthesis was carried out analogously to Example 25. The compound has the following phase sequence:

X_1 55 X_2 71 (62) S_2 67 S_A 78 N 85 I

Example 32

5-(4-Cyclohexylphenyl)-2-octyloxypyrimidine



2 g (6.96 mmol) of 5-bromo-2-octylpyrimidine, 1.42 g (6.96 mmol) of 4-cyclohexylbenzeneboronic acid, 1.5 g (14.00 mmol) of sodium carbonate and 0.08 g (0.07 mmol) of tetrakisphenylphosphine paradium were refluxed for 4 hours in 40 ml of toluene, 10 ml of ethanol and 10 ml of water.

The organic phase was separated and evaporated to dryness, and the crude product was purified by column chromatography and recrystallized from acetanilide to give

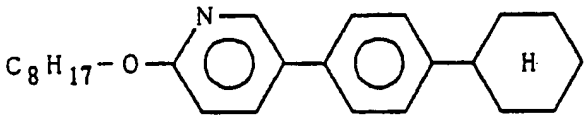
1.29 of the titled compound.

The compound has the following phase sequence:

X 92.7 (65.4) S_A 79 I

Example 33

5-(4-Cyclohexylphenyl)-2-octyloxypyridine



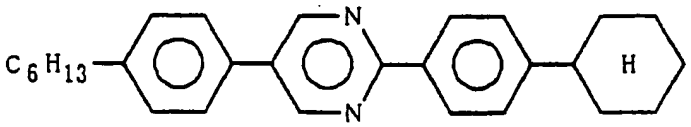
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 64 (36.1) S_B 60 I

Example 34

2-(4-Cyclohexylphenyl)-5-(4-hexylphenyl)pyrimidine



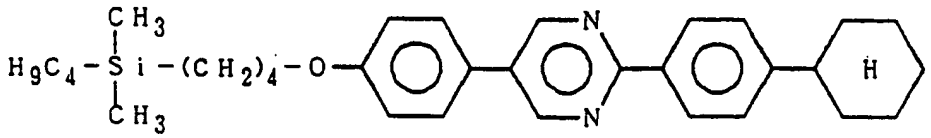
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 129.8 (115) S_3 150 S_C 153 S_A 187 N 197 I

Example 35

5-[4-(4-Butyldimethylsilyl)-butyloxyphenyl]-2-(4-cyclohexylphenyl)pyrimidine



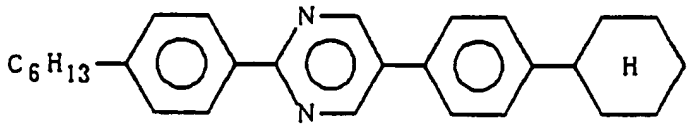
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 145.7 (140) S_1 152 I

Example 36

5-(4-Cyclohexylphenyl)-2-(4-hexylphenyl)pyrimidine



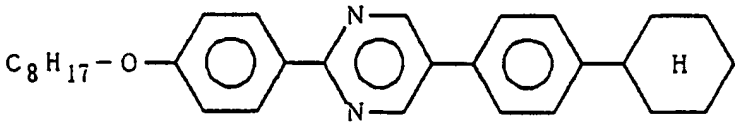
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 147 (133) S_A 209 I

Example 37

5-(4-Cyclohexylphenyl)-2-(4-octyloxyphenyl)pyrimidine



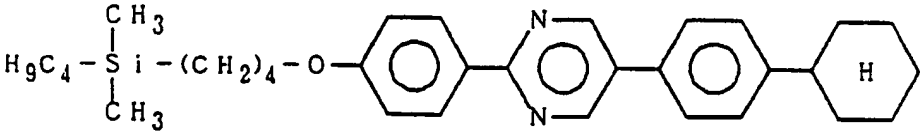
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 134 (90) S_3 94 S_C 134 S_A 210 N 219 I

Example 38

2-[4-(4-Butyldimethylsilyl)butyloxyphenyl]-5-(4-cyclohexyl-phenyl)pyrimidine



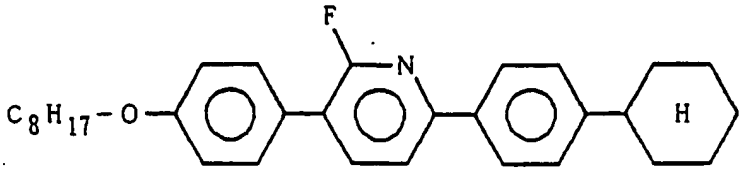
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 97 (70) S_C 117 S_A 199 I

Example 39

6-(4-Cyclohexylphenyl)-2-fluoro-3-(4-octyloxyphenyl)pyridine



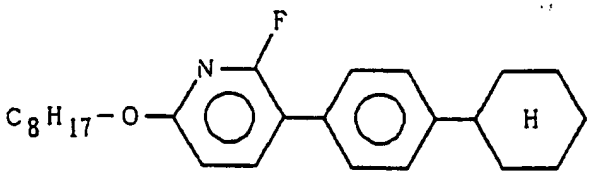
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 115 (78) S_G 99 S_C 141 N 187 I

Example 40

3-(4-Cyclohexylphenyl)-2-fluoro-6-octyloxypyridine



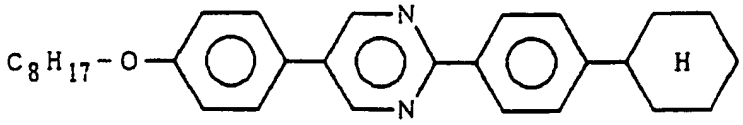
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 49 (11) I

Example 41

2-(4-Cyclohexylphenyl)-5-(4-octyloxyphenyl)pyrimidine



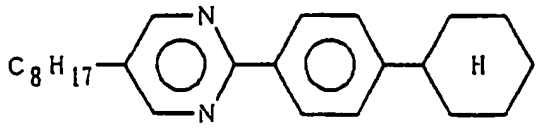
The synthesis was carried out analogously to Example 32.

The compound has the following phase sequence:

X 102 (73) S_B 144 S_C 188 S_A 214 N 217 I

Example 42

2-(4-Cyclohexylphenyl)-5-octylpyrimidine



2.9 ml (17 mmol) of 1-bromooctane and 0.51 g (21 mmol) of a Grignard compound were reacted in 50 ml of tetrahydrofuran. The Grignard compound solution was added dropwise to a solution of 3 g (9.5 mmol) of 5-bromo-2-(4-cyclohexylphenyl)-pyrimidine and 0.06 g (0.1 mmol) of 1,3-bis(diphenyl-phosphine)propane nickel (II) chloride in 65 ml of tetrahydrofuran, and the mixture was stirred at 0 °C for 2 to 4 hours.

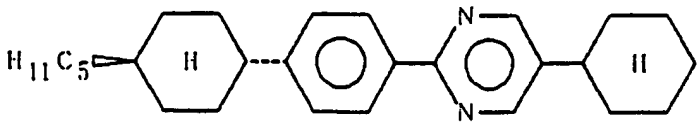
3 ml of hydrochloric acid (37 % strength) and 50 ml of water were added therein, the organic phase was separated, washed, dried over Na_2SO_4, filtered and evaporated to dryness. The product was purified by column chromatography and recrystallized from acetanilide to give 0.83 g of the titled compound.

The compound has the following phase sequence:

X_1 18.5 (5) X_2 69 S_B 76 (71) I

Example 43

Trans-5-cyclohexyl-2-[4-(4-pentylcyclohexyl)phenyl]pyrimidine



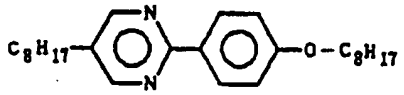
The synthesis was carried out analogously to Example 42.

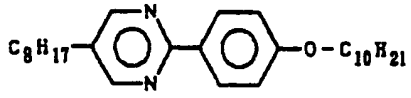
The compound has the following phase sequence:

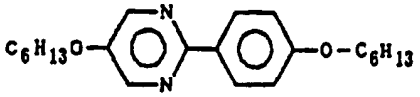
X 140 (113) N 214 I

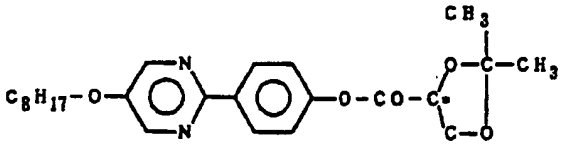
Example 44

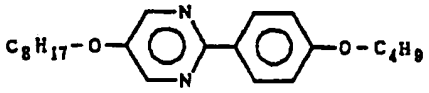
A ferroelectric liquid-crystal composition (A) containing the compounds (a) and (b) of the present invention comprises the following components (mol %).

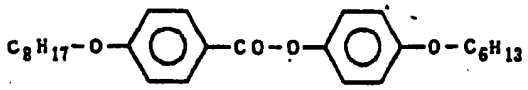
6 

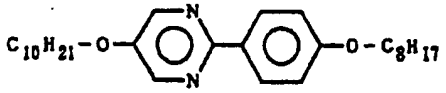
6 

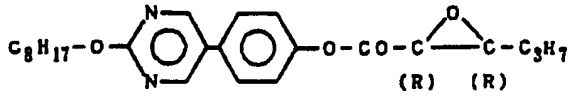
7 

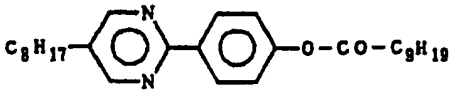
3 

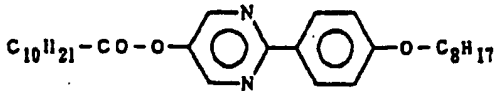
8 

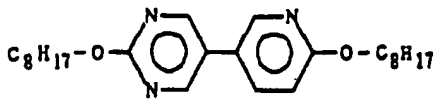
6 

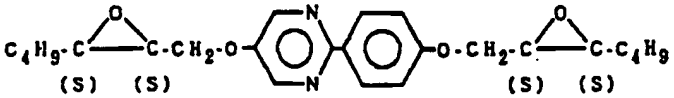
6 

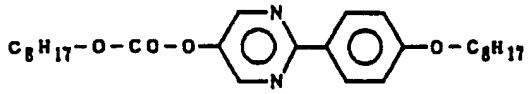
7 

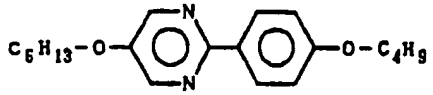
5 

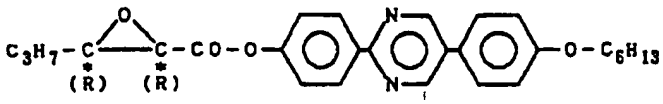
6 

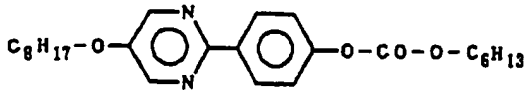
5 

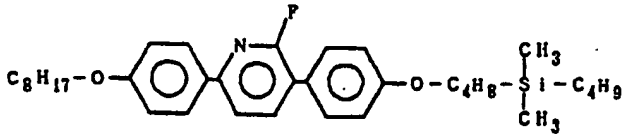
7  trans

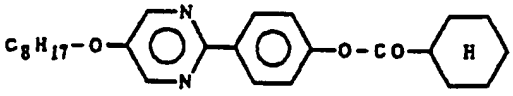
5 

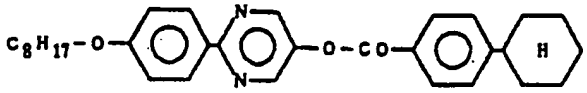
4 

4  cis

8 

7 

(a)
6 

(b)
6 

The composition (A) has the following phase sequence:

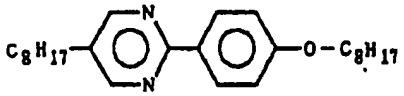
X -26 S_c* 70 S_A 73 Ch* 87 I

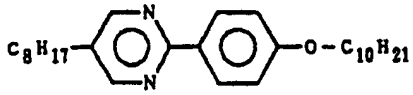
The above example shows that the compound of the present invention gives a very low melting point and a broad temperature range of smectic C phase.

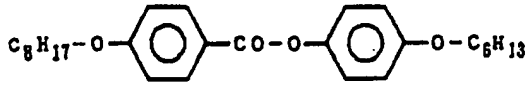
In addition, the composition (A) can be used for ferroelectric liquid-crystal display devices, and switches at a switching speed of 44 µs by means of a dipolar pulse.

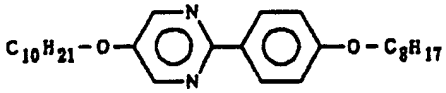
Example 45

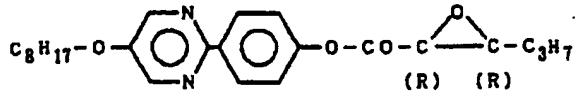
A ferroelectric liquid-crystal composition (B) containing no compounds of the present invention comprises the following components (mol %).

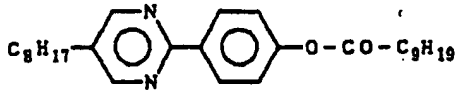
8 

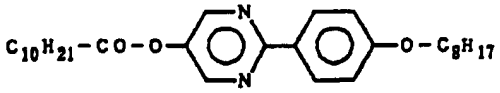
7 

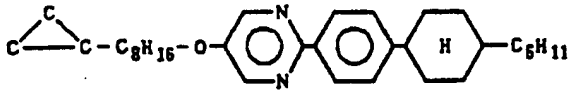
7 

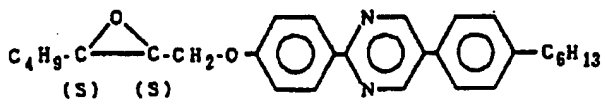
8 

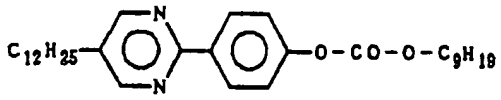
7 

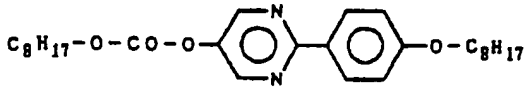
7 

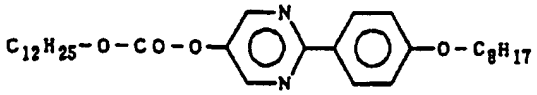
7 

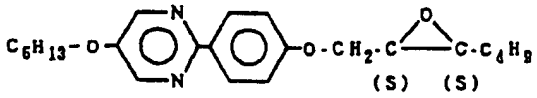
12 

8  Trans

7 

7 

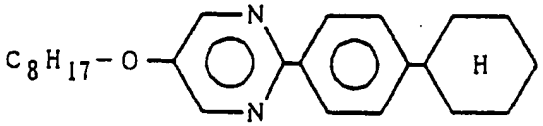
6 

9  trans

The composition (B) has the following phase sequence:

X -19 S_c* 76 S_A 85 Ch* 101 I

A ferroelectric liquid-crystal composition (C) comprising 94 % of the composition (B) and 6 % of the compound (c) of the present invention was prepared.

(c) 

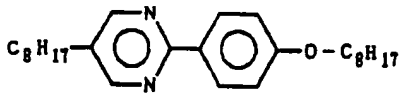
The composition (C) has the following phase sequence:

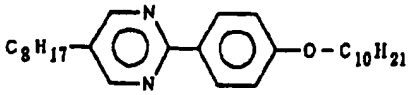
X -27 S_c* 72 S_A 85 Ch* 96 I

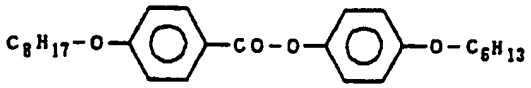
The melting point was lowered by adding the liquid-crystal compound (c) of the present invention to the ferroelectric liquid-crystal composition (B).

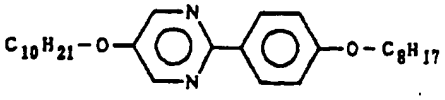
Example 46

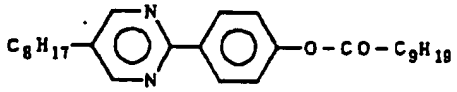
A ferroelectric liquid-crystal composition (D) containing the compounds of the present invention comprises the following components (mol %).

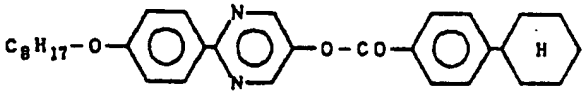
9 

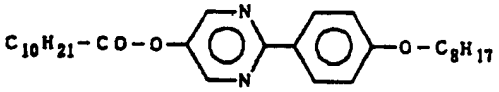
8 

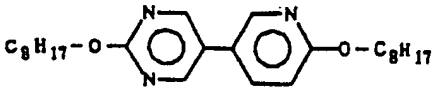
8 

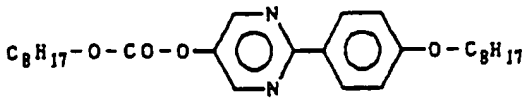
8 

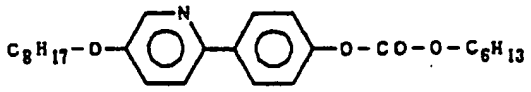
6 

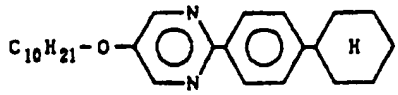
7 

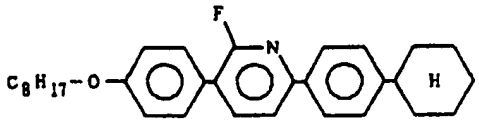
8 

6 

6 

13 

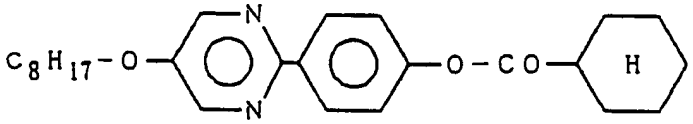
15 

6 

The composition (D) has the following phase sequence:

X -22 S_c 74 S_A 83 Ch 88 I

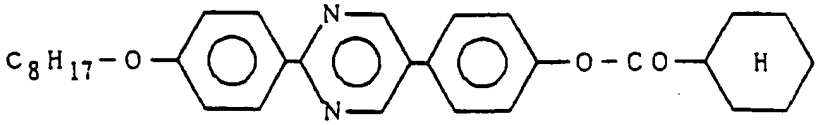
Liquid-crystal compositions (E) and (F) were prepared by adding the compounds of the present invention to the composition (D). The smectic liquid-crystal composition (E) comprises 90 % of the composition (D) and 10 % of the compound (d) of the present invention.

(d) 

The composition (E) has the following phase sequence:

X -31 S_c 75 S_A 84 Ch 97 I

On the other hand, the smectic liquid-crystal composition (F) comprises 90 % of the composition (D) and 10 % of the compound (e) of the present invention.

(e) 

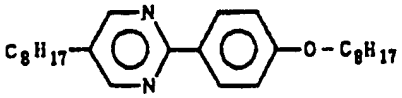
The composition (F) has the following phase sequence:

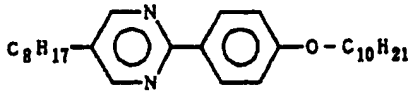
X -40 S_c 80 S_A 89 Ch 102 I

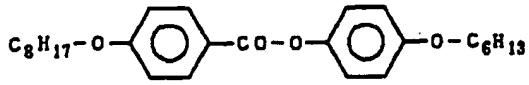
The melting point was lowered by adding the liquid-crystal compound (d) or (e) according to the present invention to the smectic liquid-crystal composition (D).

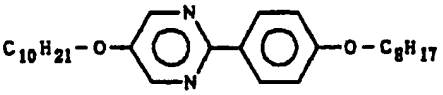
Example 47

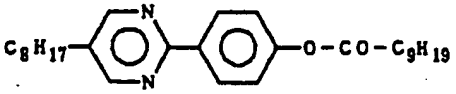
A smectic liquid-crystal composition (G) containing no compounds of the present invention comprises the following components (mol %).

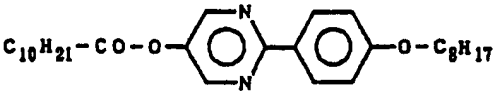
13 

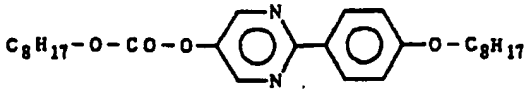
12 

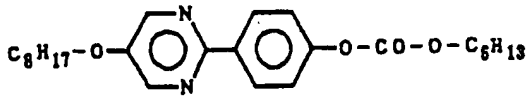
13 

12 

10 

11 

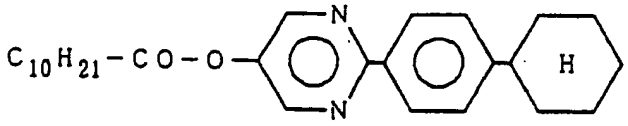
10 

19 

The composition (G) has the following phase sequence:

X -7 S_c 68 S_A 71 Ch 85 I

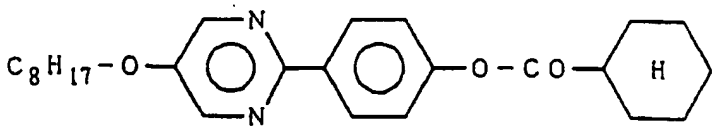
Liquid-crystal compositions (H) and (I) were prepared by adding the compound (f) or (g) of the present invention to the composition (G). The smectic liquid-crystal composition (H) comprises 90 % of the composition (G) and 10 % of the compound (f) of the present invention.

(f) 

The composition (H) has the following phase sequence:

X -13 S_c 72 S_A 77 Ch 86 I

On the other hand, the smectic liquid-crystal composition (I) comprises 80 % of the composition (G) and 20 % of the compound (g) of the present invention.

(g) 

The composition (I) has the following phase sequence:

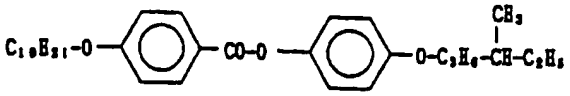
X -14 S_c 69 S_A 76 C 87 I

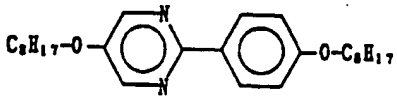
The melting point was lowered by adding the liquid-crystal compound (f) or (g) of the present invention to the smectic liquid-crystal composition (G).

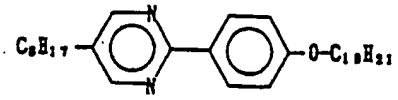
Example 48

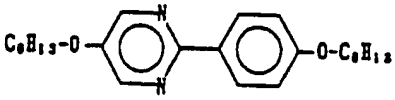
A liquid-crystal composition (J) containing no compounds of the present invention comprises the following components

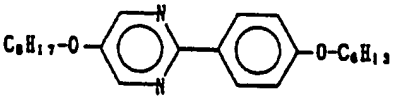
(mol %).

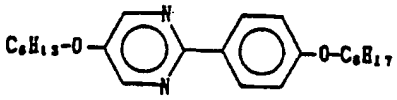
12 

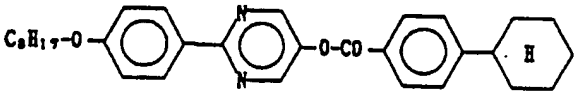
12 

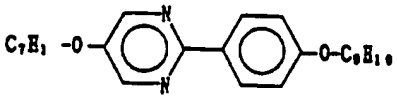
10 

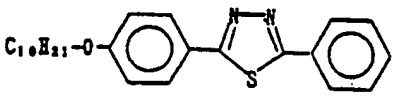
9 

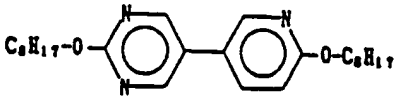
12 

12 

7 

10 

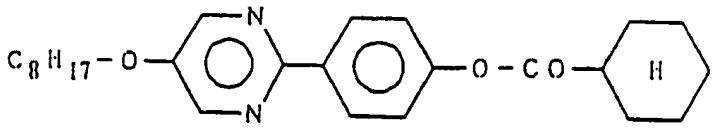
10 

6 

The composition (J) has the following phase sequence:

X 35 S_C* 71 S_A 88 Ch* 95 I

A liquid-crystal composition (K) containing 85 % of the composition (J) and 15 % of the compound (a) of the present invention was prepared.

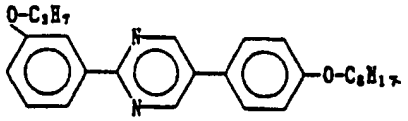
(a) 

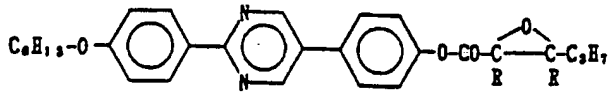
The composition (K) has the following phase sequence:

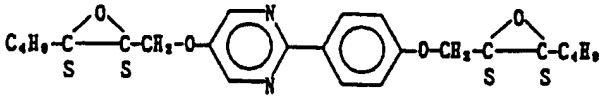
X -6 S_C 73 S_A 86 Ch 98 I

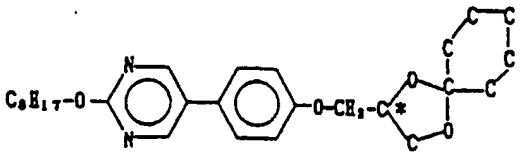
The melting point was lowered by adding the liquid-crystal compound (a) of the present invention to the liquid-crystal composition (J).

Ferroelectric liquid-crystal compositions (L) and (M) were prepared by adding the following compounds to 83 % of liquid-crystal compositions (J) and (K) respectively.

5 

7  cis

2  trans

3  S

The ferroelectric liquid-crystal compositions (L) and (M) have the following phase sequence:

(L) X -7 S_C* 73 S_A 90 Ch* 91 I

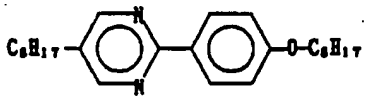
(M) X -13 S_C* 73 S_A 87 Ch* 92 I

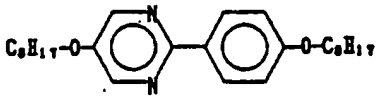
The ferroelectric liquid-crystal composition (M) has a low melting point.

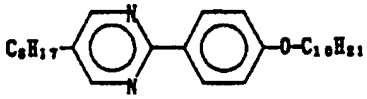
The ferroelectric liquid-crystal composition has a spontaneous polarization of 53 nC/cm^2, and it can be used for dielectric liquid-crystal display devices, and switches by means of a dipolar pulse.

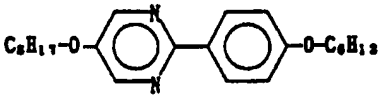
Example 49

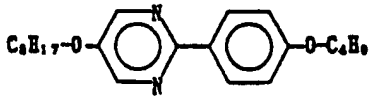
A ferroelectric liquid-crystal composition (N) containing 8 % of the compound (a) of the present invention comprises the following components (mol %).

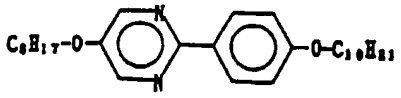
11 

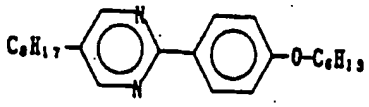
4 

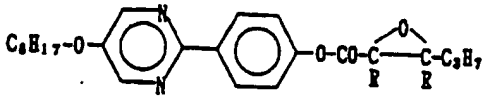
9 

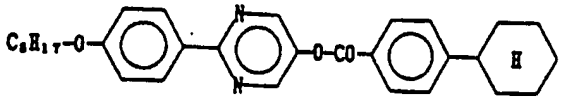
8 

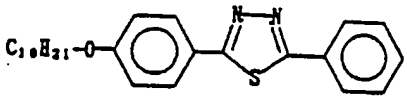
7 

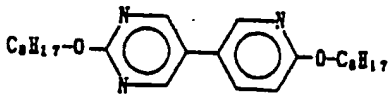
7 

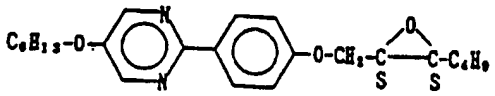
11 

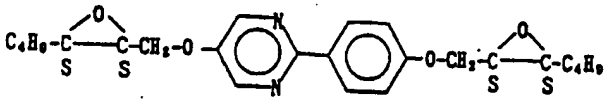
7 

4 

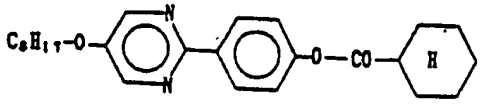
5 

4 

10  trans

5  trans

(a)

8 

The ferroelectric liquid-crystal composition (N) has the following phase sequence:

X -8 S_c* 64 S_A 80 Ch* 84 I

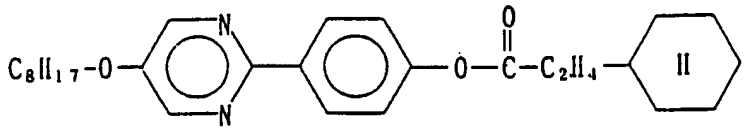
The above low melting point was accomplished by the compound of the present invention.

In addition, the ferroelectric liquid-crystal composition (N) of the present invention has a spontaneous polarization of 40 nC/cm^2, and it can be used for dielectric liquid-crystal display devices, and switches by means of a dipolar pulse.

The above Examples conform that the compounds according to the present invention can be used to prepare smectic or ferroelectric liquid-crystal compositions having a broad temperature range of a smectic C phase, particularly having a broad temperature range in a lower temperature region.

Referential Example

4-(5-Octyloxypyrimidine-2-yl)phenyl 3-cyclohexylpropionate



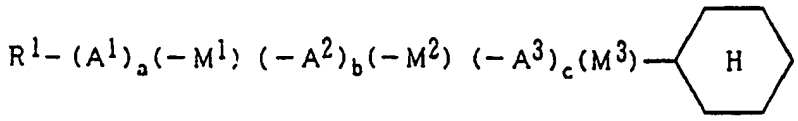
The synthesis was carried out analogously to Example 15.

The compound has the following phase sequence:

X 100 I

What is claimed is:

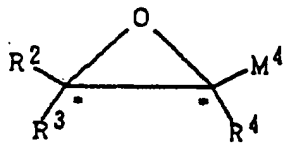
1. A compound of the formula (I):

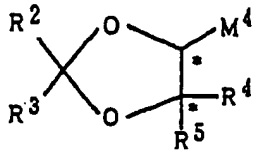


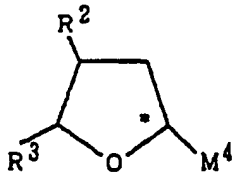
(I)

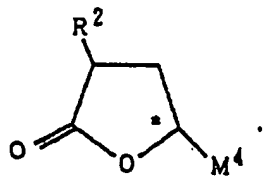
in which the symbols and indices have the following meanings:

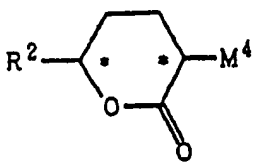
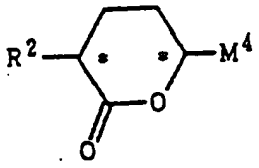
R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, and in which, in addition, one or more hydrogen atoms of the alkyl radical may be substituted by F, Cl, Br or CN, or is one of the chiral groups below:

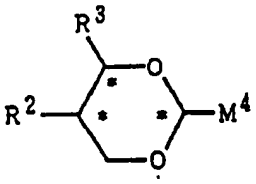
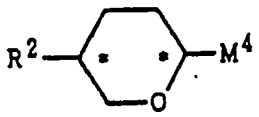


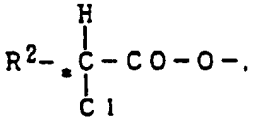
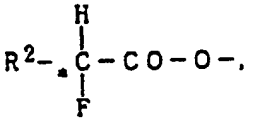


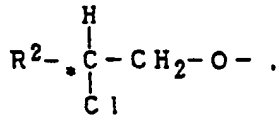
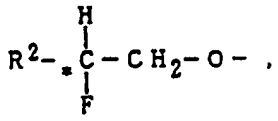


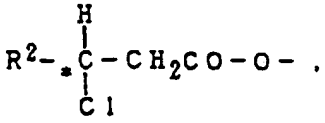
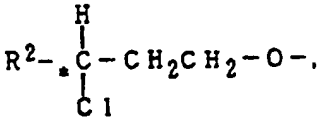


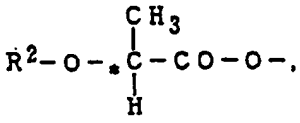
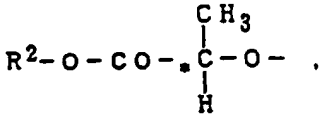



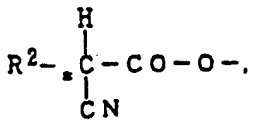
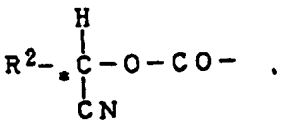



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wherein,

R^2, R^3, R^4 and R^5, independently of one another, are H or a straight-chain or branched alkyl radical having 1 to 22 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, or R^2 and R^3 together may alternatively be $-(CH_2)_4-$ or $-(CH_2)_5-$ if they are bonded as substituents to a dioxolane system; and

M^4 is $-CH_2-O-$, $-O-CH_2-$, -CO-O-, -O-CO- or a single bond;

A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, in which one or two hydrogen atoms may be replaced by F, Cl and/or CN, pyrazine-2,5-diyl, pyridazine-3,6-diyl, pyridine-2,5-diyl or pyrimidine-2,5-diyl, in which one or two hydrogen atoms may be replaced by F, trans-1,4-cyclohexylene, in which one or two hydrogen atoms may be replaced by -CN and/or $-CH_3$, 1,3,4-thiadiazole-2,5-diyl, 1,3-dioxane-2,5-diyl, 1,3-dithiane-2,5-diyl, 1,3-thiazole-2,4-diyl, 1,3-thiazole-2,5-diyl, thiophene-2,4-diyl, thiophene-2,5-diyl, piperazine-1,4-diyl, piperazine-2,5-diyl or naphthalene-2,6-diyl;

M^1 and M^2 are identical or different and are a single bond, -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

M^3 is a single bond or a straight-chain or branched alkyl radical having 1 to 16 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C- or $-Si(CH_3)_2-$, and in which, in addition, one or more hydrogen atoms of the alkyl radical may be substituted by F, Cl, Br or CN, with the proviso that M^3 is not $-O-CO-CH_2CH_2-$;

a, b and c are zero or one, with the proviso that the sum

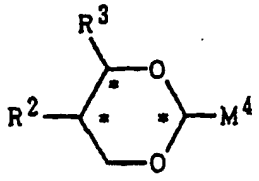
a+b+c is 2 or 3; and

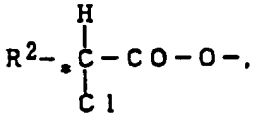
* is a chiral center;

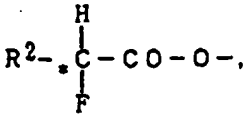
provided however that when A^1 and A^2 are 1,4-phenylene, M^1 and M^2 are a single bond, c is zero and M^3 is -CO-O-, then R^1 is not C_8H_{17}-O-.

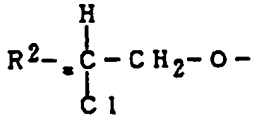
2. A compound as claimed in Claim 1, in which the symbols and indices in the formula (I) have the following meanings:

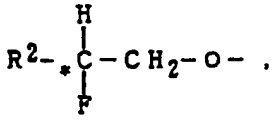
R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, or is one of the chiral groups below:

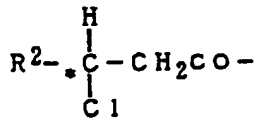


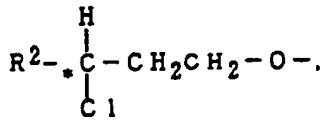












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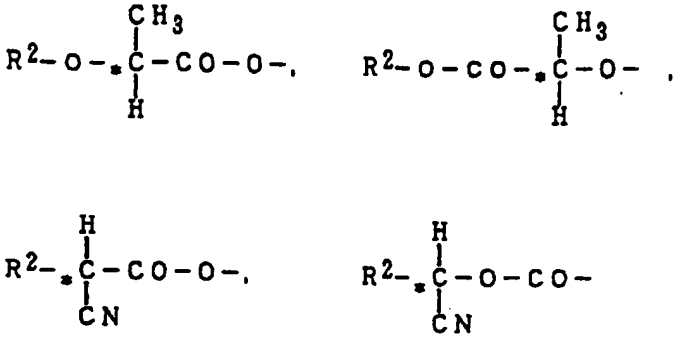


wherein,

R^2, R^3, R^4 and R^5, independently of one another, are H or a straight-chain or branched alkyl radical having 1 to 22 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -S-, -CO-, -CO-O-, -O-CO-, -CO-S-, -S-CO-, -O-CO-O-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$, or R^2 and R^3 together may alternatively be $-(CH_2)_4-$ or $-(CH_2)_5-$ if they are bonded as substituents to a dioxolane system;

A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, pyrazine-2,5-diyl, pyridazine-3,6-diyl, pyridine-2,5-diyl or pyrimidine-2,5-diyl, in which one or two hydrogen atoms may be replaced by F, trans-1,4-cyclohexylene, 1,3,4-thiadiazole-2,5-diyl, 1,3-dioxane-2,5-diyl or naphthalene-2,6-diyl;

M^1 and M^2 are identical or different and are a single bond, -O-, -CO-, -CO-O-, -O-CO-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

M^3 is a single bond or a straight-chain or branched alkyl radical having 1 to 16 carbon atoms in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -CO-, -CO-O-, -O-CO- or -O-CO-O-, and in which, in addition, one or more hydrogen atoms of the alkyl

radical may be substituted by F, Cl, Br or CN; and

a, b and c are zero or one, with the proviso that the sum a+b+c is 2 or 3.

3. A compound as claimed in Claim 1, in which the symbols and indices in the formula (I) have the following meanings:

R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms (with or without an asymmetrical carbon atom) in which, in addition, it is possible for one or two non-adjacent $-CH_2-$ groups to be replaced by -O-, -CO-, -CO-O-, -O-CO-, -CH=CH-, -C≡C-,  , or $-Si(CH_3)_2-$;

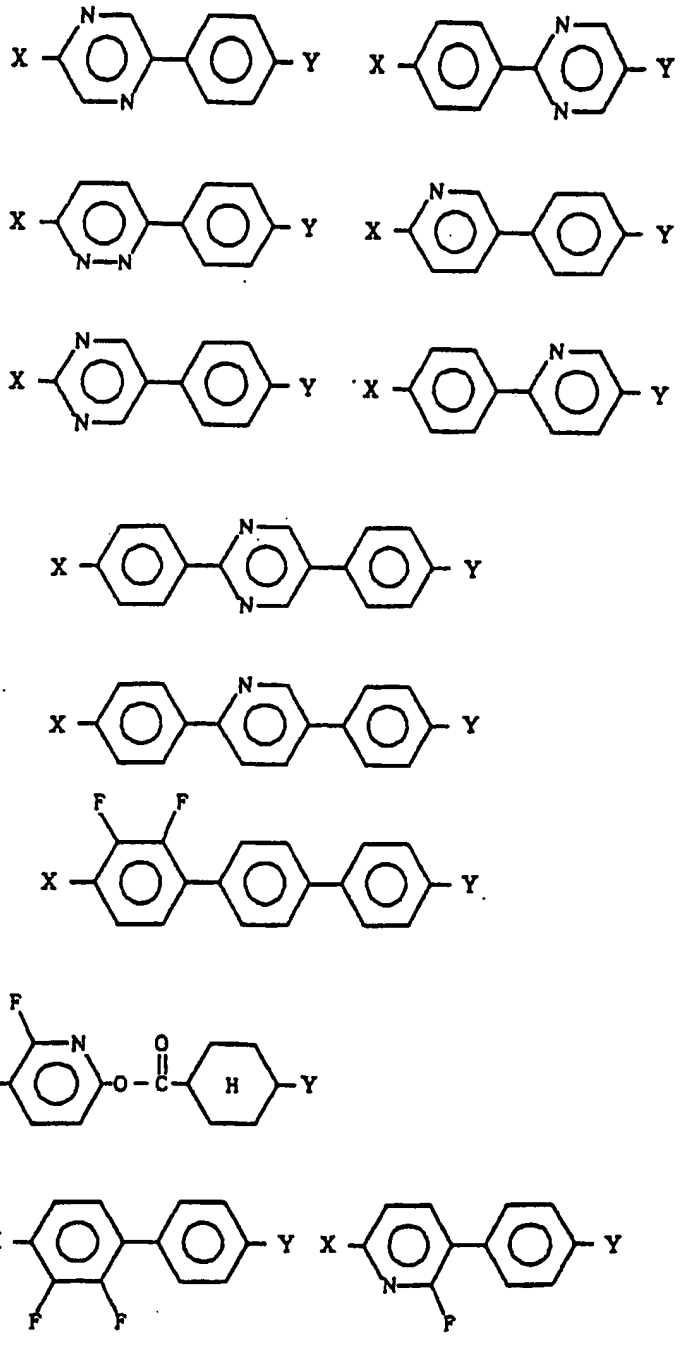
A^1, A^2 and A^3 are identical or different and are 1,4-phenylene, in which one or two hydrogen atoms may be replaced by F, pyrazine-2,5-diyl, pyridine-2,5-diyl, pyrimidine-2,5-diyl, trans-1,4-cyclohexylene, 1,3,4-thiadiazole-2,5-diyl, naphthalene-2,6-diyl or 1,3-dioxane-2,5-diyl;

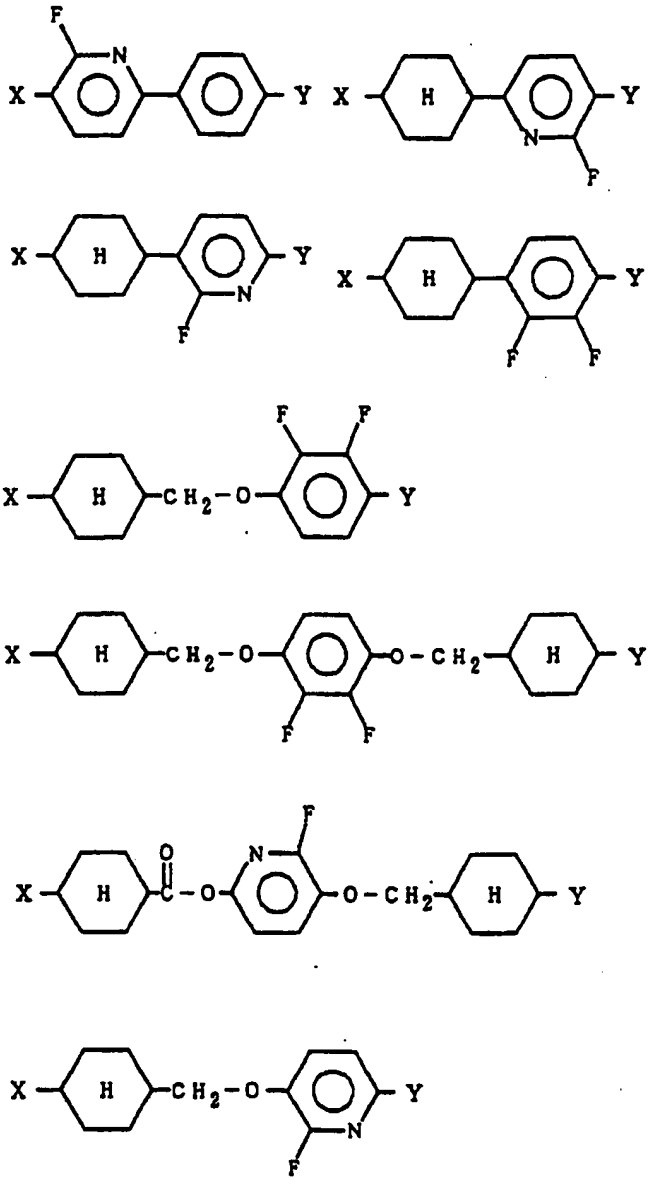
M^1 and M^2 are identical or different and are a single bond, -O-, -CO-O-, -O-CO-, $-CH_2-O-$, $-O-CH_2-$, $-CH_2CH_2-$, -CH=CH- or -C≡C-;

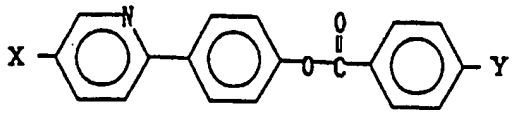
M^3 is a single bond, -CO-O-, -O-CO-, $-O-CO-C_nH_{2n}-$ or $-O-C_nH_{2n}$ (wherein n is an integer from 1 to 10); and

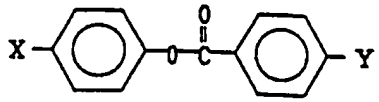
a, b and c are zero or one, with the proviso that the sum a+b+c is 2 or 3.

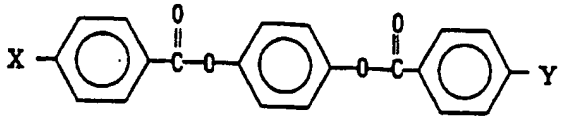
4. A compound as claimed in Claim 1 selected from the group consisting of the following compounds:

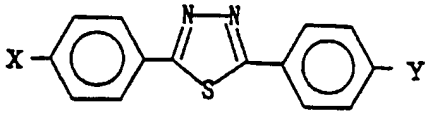


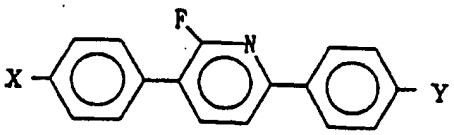


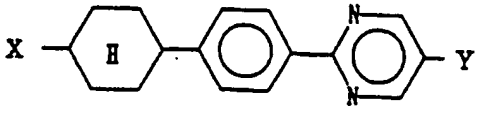


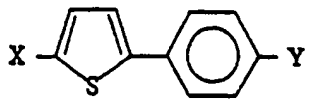


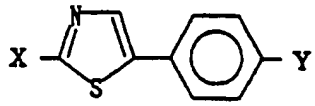


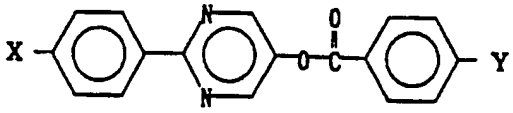


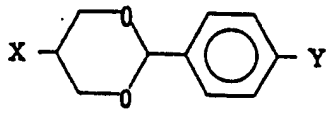


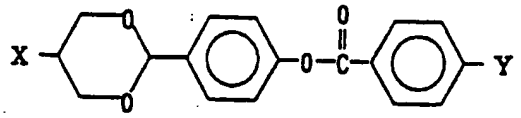


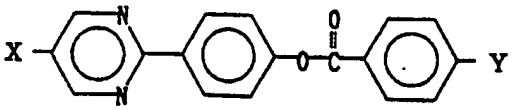






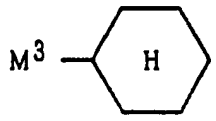






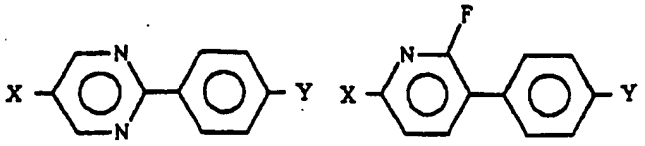
in which either of X and Y is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, wherein one $-CH_2-$ group may be replaced by -O-,  , -CH=CH-, -CO-O- or $-Si(CH_3)_2-$; and

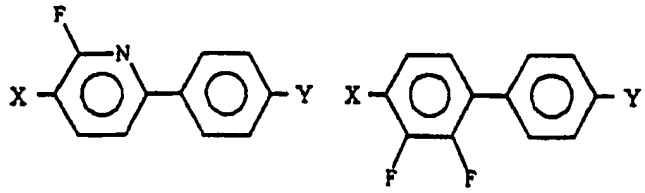
the other is

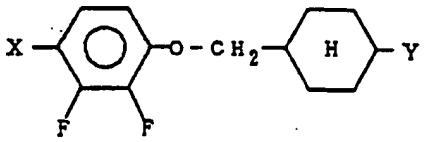


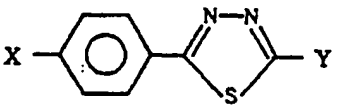
wherein M^3 is a single bond, -CO-O-, -O-CO-, $-O-CO-C_nH_{2n}-$ or $-O-C_nH_{2n}-$ (wherein n is an integer from 1 to 10).

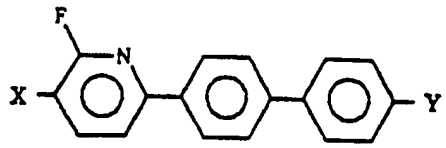
5. A compound as claimed in Claim 1 selected from the group consisting of the following compounds:

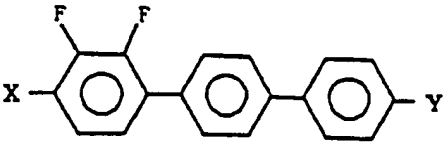


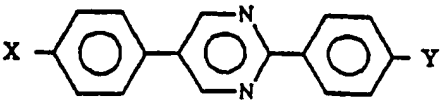






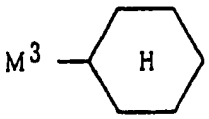






in which either of X and Y is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, wherein one $-CH_2-$ group may be replaced by -O- or -CO-O-; and

the other is

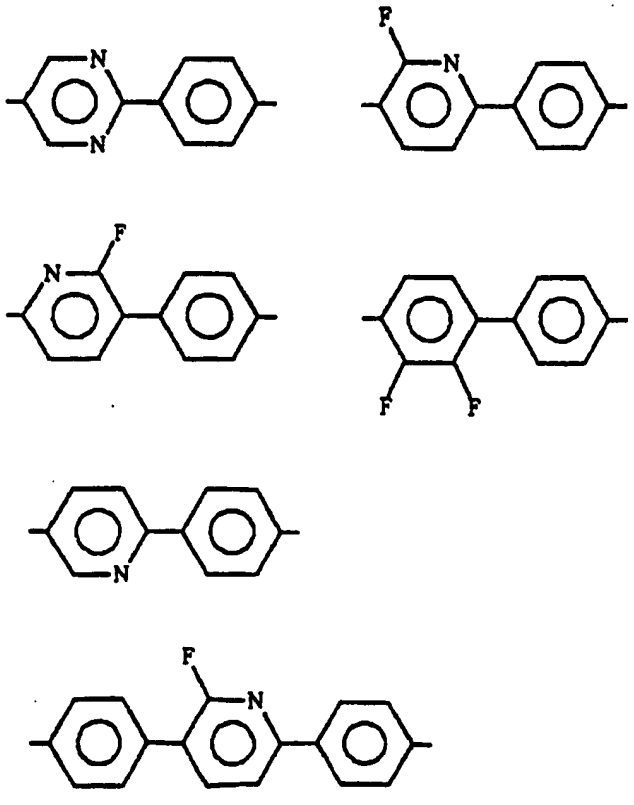


wherein M^3 is -O-CO- or $-O-CH_2-$.

6. A compound as claimed in Claim 1, in which the symbols and indices in the formula (I) have the following meanings:

R^1 is a straight-chain or branched alkyl radical having 1 to 22 carbon atoms, in which one $-CH_2-$ group may be replaced by -O- or -CO-O-; and

the group $-(A^1)_a(-M^1)(-A^2)_b(-M^2)(-A^3)_c(-M^3)-$ is any one of the following groups:



7. A smectic liquid-crystal composition or a ferroelectric liquid-crystal composition containing at least one compound as claimed in Claim 1.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP 94/01397

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 C07D239/04 C07D213/04 C07D285/12 C09K19/34 C09K19/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E	EP,A,0 606 090 (HOECHST) 13 July 1994 see page 4, line 30 - page 5, line 46 see page 13, line 15 - line 51 see page 24 - page 26 see examples 31,50-75 ---	1-7
X,P	EP,A,0 541 081 (HOECHST) 12 May 1993 see page 12, line 44 - page 13, line 10 see examples 12,13,19-23 see claims 1-7 ---	1-7
X,P	EP,A,0 552 658 (HOECHST) 28 July 1993 see page 5, line 47 - page 6, line 19 see examples 1,2,13 see claims 1-6 --- -/--	1-7

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
2 September 1994	22.09.94

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Boulon, A

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP 94/01397

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	EP,A,0 573 878 (HOECHST) 15 December 1993 see page 7, line 5 - line 27 see page 27 - page 29; examples 4,5 see claims 1-9	1-7

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/EP 94/01397

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-0606090	13-07-94	NONE		
EP-A-0541081	12-05-93	CA-A-	2082304	08-05-93
EP-A-0552658	28-07-93	NONE		
EP-A-0573878	15-12-93	AU-B-	4324193	04-01-94
		WO-A-	9325529	23-12-93
		JP-A-	6073006	15-03-94